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3-(1-{[1-(4-Bromophenyl)-1*H*-1,2,3-triazol-4-yl]-methyl}piperidin-4-yl)-6-fluoro-1,2-benzoxazole hemihydrate

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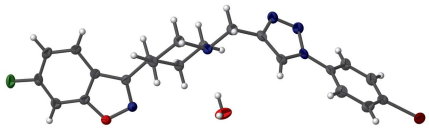
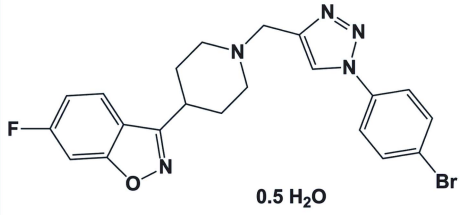
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{21}H_{19}BrFN_5O\cdot0.5H_2O$, crystallizes as a hemihydrate with the water molecule located on a twofold rotation axis. The piperidine ring has a chair conformation, whereas the triazole and the benzisoxazole rings are planar (r.m.s. deviations = 0.006 and 0.009 Å, respectively). The N—C and C—C bonds connecting the triazole and benzisoxazole rings, respectively, to the piperidine ring lie in equatorial positions. In the crystal, molecules related by a twofold rotation axis are linked *via* O—H···N hydrogen bonds involving the water molecule and a pair of C—H···N hydrogen bonds forming dimers. The dimers are linked *via* a pair of C—H···F hydrogen bonds leading to the formation of chains propagating along [101].

3D view



Chemical scheme



Structure description

Benzisoxazoles display a wide spectrum of biological activities namely antipsychotic, antitumor (Jain & Kwon, 2003), anticonvulsant, antimicrobial (Priya *et al.*, 2005), anti-thrombotic and cholinesterase inhibiting (Alzheimer's disease) properties (Rangappa & Basappa, 2005). In addition the benzisoxazole nucleus is found in a large number of pharmaceutical products, and is used in prodrugs. 1,2,4-Triazole and its derivatives belong to a class of exceptionally active compounds possessing a wide spectrum of biological properties, including anti-inflammatory, antifungal, antiviral, analgesic, anticonvulsant, antiparasitic and antidepressant activities (Naveen *et al.*, 2006). Some triazole derivatives are also known to exhibit anticancer activity. 1,2,4-Triazoles have also been investigated for their *CNS* depressant, pesticidal, antimycobacterial, hypoglycemic, diuretic, insecti-

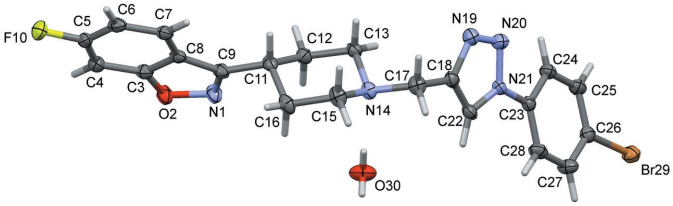


Figure 1
A view of the molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

cidal and herbicidal effects (Benaka Prasad *et al.*, 2007). In view of the above and as a part of our ongoing research on novel heterocyclic compounds, the title compound was synthesized by click reaction with good yield.

The title compound, Fig. 1, crystallizes as a hemihdyrate with the water molecule located on a twofold rotation axis. The piperidine ring has a chair conformation whereas the triazole and the benzisoxazole rings are planar (r.m.s. deviations are 0.006 and 0.009 Å, respectively). The bonds N4—C17 and C11—C9 connecting the triazole and the benzisoxazole rings, respectively, lie in equatorial positions on the piperidine ring.

In the crystal, molecules related by a twofold rotation axis are linked *via* O—H···N hydrogen bonds, involving the water molecule, and a pair of C—H···N hydrogen bonds forming dimers. The dimers are linked *via* a pair of C—H···F hydrogen bonds leading to the formation of chains propagating along [101]; see Table 1 and Fig. 2.

Synthesis and crystallization

To the stirred solution of 1-azido bromobenzene (2 mmol) and alkyne (2 mmol) in 2 ml of acetonitrile and 1 ml of water, copper iodide (0.2 mmol) was added at ambient temperature. The reaction mixture was stirred at room temperature for 4–8 h (monitored by TLC). On completion of the reaction, the mixture was poured into ice cold water (10 ml), extracted with ethyl acetate (3 × 10 ml). The combined organic layer was washed with water (10 ml), followed by brine (10 ml) and dried over anhydrous sodium sulfate, concentrated under reduced pressure to get the crude triazole, which was purified by column chromatography over silica gel (60–120 mesh) using hexane:EtOAc (8:2) as eluent. Good quality single crystals were obtained in good yield by slow evaporation of the solvent. ^{1}H NMR(400 MHz, DMSO-*d*6, p.p.m.): δ = 7.94 (*s*, 1H),7.72–7.69 (*m*, 1H, Ar—H), 7.66–7.61 (*m*, 4H, Ar—H), 7.24–7.00 (*m*, 1H, Ar—H), 3.81 (*s*, 2H), 3.14–3.09 (*m*, 4H), 2.33–2.31 (*m*,1*H*), 2.22–2.09 (*m*, 4H). ^{13}C NMR (400 MHz, DMSO-d_6, p.p.m.): δ = 160.4, 156.5, 148.2, 136.2, 133.2, 129.2, 128.0, 123.7, 123.1, 119.2, 117.5, 111.3, 57.6, 54.1, 30.1, 27.5. LCMS (*MM*: ES + APCL) 456.10, 458.10 $(M + H)^+$. HPLC purity = 95%. Anal. calc. for $C_{21}H_{19}N_5O_3FBr$: C, 55.27; H, 4.20; N, 15.35%; found: C, 56.15; H, 4.03; N, 15.88%.

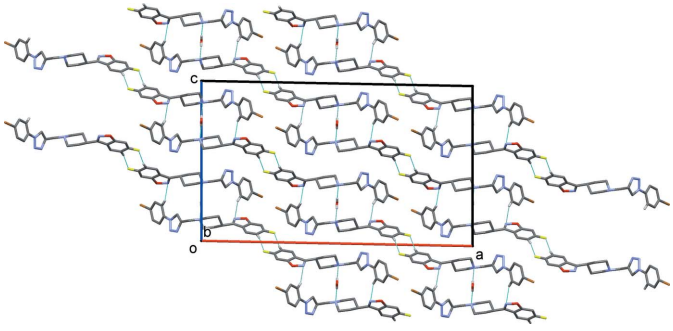


Figure 2
A view in projection along the *b* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1), and H atoms not involved in hydrogen bonding have been omitted for clarity.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O30—H30···N14^{i}	0.82 (3)	2.14 (3)	2.946 (3)	172 (3)
C28—H28···N1^{i}	0.93	2.56	3.430 (4)	157
C6—H6···F10^{ii}	0.93	2.49	3.394 (3)	163

Symmetry codes: (i) $-x+1, y, -z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y-\frac{1}{2}, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{19}BrFN_5O{\cdot}0.5H_2O$
M_r	465.33
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	34.4311 (10), 5.6779 (2), 20.2869 (6)
β (°)	91.107 (1)
V (Å^3)	3965.3 (2)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	3.13
Crystal size (mm)	0.30 × 0.27 × 0.25
Data collection	
Diffractometer	Bruker X8 Proteum diffractometer
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.454, 0.508
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11135, 3195, 3080
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.096, 1.10
No. of reflections	3195
No. of parameters	271
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.48, −0.45

Computer programs: *APEX2* (Bruker, 2013), *SAINT* (Bruker, 2013)', *SHELXS97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x152458 [doi:10.1107/S241431461502458X]

3-(1-{[1-(4-Bromophenyl)-1*H*-1,2,3-triazol-4-yl]methyl}piperidin-4-yl)-6-fluoro-1,2-benzoxazole hemihydrate

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3-(1-{[1-(4-Bromophenyl)-1*H*-1,2,3-triazol-4-yl]methyl}piperidin-4-yl)-6-fluoro-1,2-benzoxazole hemihydrate

Crystal data

$C_{21}H_{19}BrFN_5O·0.5H_2O$
M_r = 465.33
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 34.4311 (10) Å
b = 5.6779 (2) Å
c = 20.2869 (6) Å
β = 91.107 (1)°
V = 3965.3 (2) Å^3
Z = 8
F(000) = 1896
D_x = 1.559 Mg m^{-3}
Cu *K*α radiation, λ = 1.54178 Å
Cell parameters from 3195 reflections
θ = 5.0–64.3°
μ = 3.13 mm^{-1}
T = 296 K
Rectangle, yellow
0.30 × 0.27 × 0.25 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Rotating Anode
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.454, T_{max} = 0.508
11135 measured reflections
3195 independent reflections
3080 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 64.3°, θ_{min} = 5.0°
h = −40→38
k = −6→6
l = −22→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.096
S = 1.10
3195 reflections
271 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2 + 5.9658P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br29	0.72298 (1)	1.29638 (4)	0.28345 (1)	0.0287 (1)
F10	0.23150 (4)	0.0267 (3)	0.44145 (7)	0.0357 (5)
O2	0.33559 (5)	0.4753 (3)	0.35433 (8)	0.0283 (5)
N1	0.37653 (6)	0.4371 (4)	0.35741 (11)	0.0285 (6)
N14	0.50507 (5)	0.1278 (3)	0.36583 (10)	0.0217 (6)
N19	0.58992 (6)	0.2882 (4)	0.44413 (11)	0.0288 (7)
N20	0.61405 (6)	0.4651 (4)	0.43820 (10)	0.0284 (6)
N21	0.61341 (5)	0.5296 (3)	0.37376 (9)	0.0215 (6)
C3	0.31868 (7)	0.2941 (4)	0.38620 (12)	0.0236 (7)
C4	0.27888 (7)	0.2670 (5)	0.39576 (12)	0.0267 (8)
C5	0.26950 (7)	0.0657 (5)	0.42895 (12)	0.0265 (7)
C6	0.29596 (7)	−0.1025 (4)	0.45121 (11)	0.0270 (7)
C7	0.33528 (7)	−0.0691 (4)	0.44156 (11)	0.0237 (7)
C8	0.34652 (7)	0.1361 (4)	0.40874 (11)	0.0218 (7)
C9	0.38264 (7)	0.2405 (4)	0.38924 (12)	0.0232 (7)
C11	0.42293 (7)	0.1504 (4)	0.40130 (12)	0.0233 (7)
C12	0.45141 (7)	0.3549 (4)	0.41267 (12)	0.0238 (7)
C13	0.49289 (7)	0.2656 (4)	0.42274 (12)	0.0224 (7)
C15	0.47923 (7)	−0.0769 (4)	0.35656 (13)	0.0280 (7)
C16	0.43714 (7)	−0.0008 (4)	0.34432 (12)	0.0268 (7)
C17	0.54513 (7)	0.0420 (4)	0.37479 (13)	0.0274 (7)
C18	0.57400 (7)	0.2368 (4)	0.38362 (13)	0.0242 (7)
C22	0.58880 (7)	0.3897 (4)	0.33872 (12)	0.0243 (7)
C23	0.63796 (7)	0.7157 (4)	0.35159 (12)	0.0212 (7)
C24	0.65390 (7)	0.8705 (5)	0.39741 (12)	0.0273 (7)
C25	0.67846 (7)	1.0469 (5)	0.37636 (12)	0.0279 (7)
C26	0.68691 (7)	1.0639 (4)	0.31016 (12)	0.0237 (7)
C27	0.67048 (8)	0.9124 (4)	0.26426 (12)	0.0296 (8)
C28	0.64577 (8)	0.7368 (5)	0.28518 (13)	0.0283 (8)
O30	0.50000	0.4399 (5)	0.25000	0.0372 (9)
H4	0.26050	0.37560	0.38100	0.0320*
H6	0.28730	−0.23710	0.47250	0.0320*
H7	0.35350	−0.17880	0.45630	0.0280*
H11	0.42290	0.05300	0.44120	0.0280*
H12A	0.45030	0.45990	0.37500	0.0290*

H12B	0.44380	0.44360	0.45120	0.0290*
H13A	0.51030	0.39840	0.42900	0.0270*
H13B	0.49440	0.16860	0.46210	0.0270*
H15A	0.48070	−0.17590	0.39550	0.0340*
H15B	0.48790	−0.16910	0.31940	0.0340*
H16A	0.43520	0.08830	0.30360	0.0320*
H16B	0.42080	−0.13910	0.33970	0.0320*
H17A	0.55200	−0.05100	0.33670	0.0330*
H17B	0.54640	−0.06010	0.41310	0.0330*
H22	0.58320	0.39640	0.29370	0.0290*
H24	0.64820	0.85600	0.44180	0.0330*
H25	0.68920	1.15310	0.40650	0.0330*
H27	0.67600	0.92830	0.21980	0.0360*
H28	0.63450	0.63350	0.25480	0.0340*
H30	0.4993 (11)	0.342 (5)	0.2205 (13)	0.054 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br29	0.0303 (2)	0.0306 (2)	0.0252 (2)	−0.0042 (1)	0.0009 (1)	0.0021 (1)
F10	0.0231 (7)	0.0524 (9)	0.0319 (8)	−0.0093 (6)	0.0054 (6)	0.0010 (7)
O2	0.0230 (8)	0.0290 (9)	0.0326 (10)	−0.0011 (7)	−0.0032 (7)	0.0101 (7)
N1	0.0214 (10)	0.0314 (11)	0.0325 (12)	−0.0036 (8)	−0.0026 (9)	0.0106 (9)
N14	0.0218 (10)	0.0183 (9)	0.0249 (10)	−0.0014 (8)	−0.0012 (8)	0.0003 (8)
N19	0.0243 (11)	0.0371 (13)	0.0248 (12)	−0.0036 (9)	−0.0006 (9)	0.0003 (9)
N20	0.0283 (11)	0.0366 (11)	0.0203 (10)	−0.0035 (9)	0.0013 (8)	−0.0005 (9)
N21	0.0165 (9)	0.0287 (10)	0.0191 (10)	−0.0001 (8)	−0.0024 (7)	−0.0030 (8)
C3	0.0287 (13)	0.0241 (12)	0.0179 (12)	−0.0040 (9)	−0.0002 (10)	0.0012 (8)
C4	0.0236 (13)	0.0345 (13)	0.0220 (13)	0.0003 (10)	−0.0009 (10)	−0.0009 (10)
C5	0.0203 (12)	0.0399 (13)	0.0193 (12)	−0.0073 (10)	0.0011 (9)	−0.0039 (10)
C6	0.0321 (13)	0.0312 (13)	0.0177 (12)	−0.0113 (10)	0.0014 (10)	0.0009 (10)
C7	0.0286 (12)	0.0258 (11)	0.0165 (11)	−0.0046 (10)	−0.0027 (9)	0.0007 (9)
C8	0.0258 (12)	0.0243 (11)	0.0151 (11)	−0.0044 (10)	−0.0026 (9)	−0.0013 (9)
C9	0.0246 (13)	0.0247 (11)	0.0202 (12)	−0.0055 (10)	−0.0019 (10)	0.0015 (9)
C11	0.0210 (11)	0.0258 (11)	0.0230 (12)	−0.0048 (10)	−0.0033 (9)	0.0072 (10)
C12	0.0234 (12)	0.0245 (11)	0.0235 (12)	−0.0029 (10)	−0.0006 (9)	−0.0032 (9)
C13	0.0227 (12)	0.0239 (11)	0.0205 (12)	−0.0037 (9)	−0.0028 (9)	0.0011 (9)
C15	0.0275 (12)	0.0194 (11)	0.0370 (14)	−0.0031 (9)	−0.0046 (10)	−0.0015 (10)
C16	0.0252 (12)	0.0215 (11)	0.0335 (14)	−0.0046 (9)	−0.0075 (10)	0.0005 (10)
C17	0.0264 (12)	0.0226 (11)	0.0330 (14)	0.0001 (10)	−0.0015 (10)	0.0001 (10)
C18	0.0190 (12)	0.0276 (11)	0.0258 (13)	0.0035 (9)	−0.0032 (10)	−0.0025 (10)
C22	0.0218 (12)	0.0290 (12)	0.0219 (12)	0.0014 (10)	−0.0065 (9)	−0.0027 (10)
C23	0.0172 (11)	0.0252 (12)	0.0212 (12)	0.0031 (9)	−0.0019 (9)	−0.0024 (9)
C24	0.0260 (12)	0.0371 (13)	0.0188 (12)	−0.0057 (11)	0.0002 (9)	−0.0041 (10)
C25	0.0277 (12)	0.0339 (13)	0.0220 (12)	−0.0041 (10)	−0.0005 (10)	−0.0075 (10)
C26	0.0225 (11)	0.0256 (11)	0.0228 (12)	0.0035 (9)	−0.0009 (9)	0.0007 (9)
C27	0.0383 (14)	0.0323 (13)	0.0183 (12)	−0.0019 (11)	0.0029 (10)	−0.0002 (10)
C28	0.0330 (14)	0.0311 (12)	0.0208 (13)	−0.0033 (11)	−0.0021 (11)	−0.0069 (10)

O30	0.0627 (18)	0.0260 (13)	0.0229 (14)	0.0000	0.0005 (13)	0.0000

Geometric parameters (Å, º)

Br29—C26	1.899 (2)	C17—C18	1.496 (3)
F10—C5	1.356 (3)	C18—C22	1.364 (3)
O2—N1	1.426 (3)	C23—C24	1.385 (4)
O2—C3	1.353 (3)	C23—C28	1.384 (4)
O30—H30^{i}	0.82 (3)	C24—C25	1.384 (4)
O30—H30	0.82 (3)	C25—C26	1.383 (3)
N1—C9	1.305 (3)	C26—C27	1.381 (3)
N14—C13	1.463 (3)	C27—C28	1.383 (4)
N14—C17	1.471 (3)	C4—H4	0.9300
N14—C15	1.474 (3)	C6—H6	0.9300
N19—C18	1.366 (3)	C7—H7	0.9300
N19—N20	1.311 (3)	C11—H11	0.9800
N20—N21	1.357 (3)	C12—H12B	0.9700
N21—C22	1.353 (3)	C12—H12A	0.9700
N21—C23	1.431 (3)	C13—H13B	0.9700
C3—C4	1.396 (3)	C13—H13A	0.9700
C3—C8	1.384 (3)	C15—H15A	0.9700
C4—C5	1.368 (4)	C15—H15B	0.9700
C5—C6	1.389 (4)	C16—H16B	0.9700
C6—C7	1.385 (3)	C16—H16A	0.9700
C7—C8	1.400 (3)	C17—H17B	0.9700
C8—C9	1.440 (3)	C17—H17A	0.9700
C9—C11	1.494 (3)	C22—H22	0.9300
C11—C16	1.528 (3)	C24—H24	0.9300
C11—C12	1.534 (3)	C25—H25	0.9300
C12—C13	1.526 (3)	C27—H27	0.9300
C15—C16	1.528 (3)	C28—H28	0.9300
N1—O2—C3	107.31 (18)	C26—C27—C28	119.3 (2)
H30—O30—H30^{i}	94 (3)	C23—C28—C27	119.6 (2)
O2—N1—C9	107.55 (19)	C5—C4—H4	123.00
C13—N14—C15	109.94 (18)	C3—C4—H4	123.00
C15—N14—C17	108.48 (17)	C5—C6—H6	120.00
C13—N14—C17	111.23 (19)	C7—C6—H6	120.00
N20—N19—C18	109.0 (2)	C6—C7—H7	121.00
N19—N20—N21	107.26 (19)	C8—C7—H7	121.00
N20—N21—C23	120.21 (18)	C12—C11—H11	108.00
C22—N21—C23	129.5 (2)	C16—C11—H11	108.00
N20—N21—C22	110.24 (18)	C9—C11—H11	108.00
O2—C3—C8	110.4 (2)	C11—C12—H12A	109.00
C4—C3—C8	123.9 (2)	C13—C12—H12A	109.00
O2—C3—C4	125.7 (2)	C13—C12—H12B	109.00
C3—C4—C5	113.7 (2)	C11—C12—H12B	109.00
F10—C5—C6	117.1 (2)	H12A—C12—H12B	108.00

C4—C5—C6	125.1 (2)	N14—C13—H13B	110.00
F10—C5—C4	117.8 (2)	C12—C13—H13A	109.00
C5—C6—C7	119.8 (2)	C12—C13—H13B	110.00
C6—C7—C8	117.5 (2)	H13A—C13—H13B	108.00
C3—C8—C7	120.0 (2)	N14—C13—H13A	109.00
C3—C8—C9	103.8 (2)	N14—C15—H15B	109.00
C7—C8—C9	136.2 (2)	C16—C15—H15A	109.00
N1—C9—C11	121.0 (2)	N14—C15—H15A	109.00
C8—C9—C11	128.2 (2)	H15A—C15—H15B	108.00
N1—C9—C8	110.9 (2)	C16—C15—H15B	109.00
C9—C11—C16	112.2 (2)	C11—C16—H16A	109.00
C12—C11—C16	109.02 (19)	C11—C16—H16B	110.00
C9—C11—C12	110.74 (19)	C15—C16—H16B	109.00
C11—C12—C13	111.26 (19)	H16A—C16—H16B	108.00
N14—C13—C12	110.73 (19)	C15—C16—H16A	110.00
N14—C15—C16	111.48 (18)	N14—C17—H17B	109.00
C11—C16—C15	110.6 (2)	C18—C17—H17A	109.00
N14—C17—C18	112.92 (18)	C18—C17—H17B	109.00
N19—C18—C17	121.3 (2)	H17A—C17—H17B	108.00
N19—C18—C22	108.4 (2)	N14—C17—H17A	109.00
C17—C18—C22	130.3 (2)	C18—C22—H22	127.00
N21—C22—C18	105.1 (2)	N21—C22—H22	127.00
N21—C23—C24	119.1 (2)	C23—C24—H24	120.00
C24—C23—C28	121.1 (2)	C25—C24—H24	120.00
N21—C23—C28	119.8 (2)	C26—C25—H25	120.00
C23—C24—C25	119.3 (2)	C24—C25—H25	120.00
C24—C25—C26	119.4 (2)	C26—C27—H27	120.00
Br29—C26—C27	120.16 (18)	C28—C27—H27	120.00
C25—C26—C27	121.4 (2)	C23—C28—H28	120.00
Br29—C26—C25	118.47 (18)	C27—C28—H28	120.00

N1—O2—C3—C4	178.5 (2)	C5—C6—C7—C8	−0.5 (3)
N1—O2—C3—C8	−1.4 (3)	C6—C7—C8—C3	−1.2 (3)
C3—O2—N1—C9	0.6 (2)	C6—C7—C8—C9	179.3 (3)
O2—N1—C9—C8	0.4 (3)	C3—C8—C9—N1	−1.1 (3)
O2—N1—C9—C11	−179.7 (2)	C3—C8—C9—C11	178.9 (2)
C15—N14—C13—C12	−60.2 (2)	C7—C8—C9—N1	178.4 (3)
C17—N14—C13—C12	179.63 (18)	C7—C8—C9—C11	−1.6 (5)
C13—N14—C15—C16	60.3 (3)	N1—C9—C11—C12	34.8 (3)
C17—N14—C15—C16	−177.9 (2)	N1—C9—C11—C16	−87.3 (3)
C13—N14—C17—C18	−59.9 (3)	C8—C9—C11—C12	−145.3 (2)
C15—N14—C17—C18	179.1 (2)	C8—C9—C11—C16	92.7 (3)
C18—N19—N20—N21	0.6 (3)	C9—C11—C12—C13	−178.1 (2)
N20—N19—C18—C17	−179.9 (2)	C16—C11—C12—C13	−54.1 (3)
N20—N19—C18—C22	−0.4 (3)	C9—C11—C16—C15	176.53 (19)
N19—N20—N21—C22	−0.6 (3)	C12—C11—C16—C15	53.5 (2)
N19—N20—N21—C23	−178.12 (19)	C11—C12—C13—N14	58.2 (2)
N20—N21—C22—C18	0.3 (3)	N14—C15—C16—C11	−57.5 (3)

C23—N21—C22—C18	177.6 (2)	N14—C17—C18—N19	104.5 (3)
N20—N21—C23—C24	−17.8 (3)	N14—C17—C18—C22	−74.9 (3)
N20—N21—C23—C28	161.4 (2)	N19—C18—C22—N21	0.1 (3)
C22—N21—C23—C24	165.2 (2)	C17—C18—C22—N21	179.5 (2)
C22—N21—C23—C28	−15.6 (4)	N21—C23—C24—C25	178.3 (2)
O2—C3—C4—C5	179.2 (2)	C28—C23—C24—C25	−0.9 (4)
C8—C3—C4—C5	−1.0 (4)	N21—C23—C28—C27	−178.0 (2)
O2—C3—C8—C7	−178.1 (2)	C24—C23—C28—C27	1.3 (4)
O2—C3—C8—C9	1.5 (3)	C23—C24—C25—C26	−0.6 (4)
C4—C3—C8—C7	2.1 (4)	C24—C25—C26—Br29	−176.49 (19)
C4—C3—C8—C9	−178.3 (2)	C24—C25—C26—C27	1.8 (4)
C3—C4—C5—F10	178.9 (2)	Br29—C26—C27—C28	176.8 (2)
C3—C4—C5—C6	−0.8 (4)	C25—C26—C27—C28	−1.5 (4)
F10—C5—C6—C7	−178.2 (2)	C26—C27—C28—C23	−0.1 (4)
C4—C5—C6—C7	1.6 (4)		

Symmetry code: (i) $-x+1, y, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O30—H30···N14^{i}	0.82 (3)	2.14 (3)	2.946 (3)	172 (3)
C28—H28···N1^{i}	0.93	2.56	3.430 (4)	157
C6—H6···F10^{ii}	0.93	2.49	3.394 (3)	163

Symmetry codes: (i) $-x+1, y, -z+1/2$; (ii) $-x+1/2, -y-1/2, -z+1$.